Microcalorimeter EDS with 3 eV energy resolution

D. A. Wollman†, K. D. Irwin†, G. C. Hilton†, L. L. Dulcie†, N. F. Bergren†, Dale E. Newbury‡, and John M. Martinis†

† National Institute of Standards and Technology (NIST), Boulder, CO 80303 USA
‡ National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899 USA

We believe that a revolutionary advance in x-ray microanalysis will occur in the next few years due to the commercial development of the NIST microcalorimeter energy dispersive spectrometer (EDS), which combines the excellent energy resolution of a wavelength dispersive spectrometer (WDS) with the parallel energy detection of an EDS. The energy resolution of microcalorimeter EDS (3 eV to 10 eV) is now comparable to that of WDS; future microcalorimeter detectors may reach fundamental energy resolution limits as low as 0.5 eV to 1 eV.

In a microcalorimeter, the energy of an x-ray is converted into heat, and a measurement of the temperature rise of the detector gives the deposited photon energy. Our microcalorimeter EDS consists of a superconducting transition-edge thermometer cooled to an operating temperature of 100 mK by a compact adiabatic refrigerator, a read-out SQUID (Superconducting Quantum Interference Device) preamplifier followed by pulse-shaping amplifiers and pile-up rejection circuitry, and a multi-channel analyzer with real-time computer interface.[1] The detector is located directly behind 4 K infrared-blocking x-ray windows and a vacuum window at the end of a snout, which extends from the cryostat into a scanning electron microscope (SEM). The small area of the x-ray absorber, typically 400 μm by 400 μm, has been effectively increased through the use of an x-ray polycapillary optic placed between the x-ray source and the detector.[1] The performance of our microcalorimeter EDS then approaches that of a high-resolution semiconductor EDS in terms of effective collection area (4 mm^2) and count rate (500 s^{-1}, and over 1000 s^{-1} using a beamblanker). The excellent energy resolution of our "general purpose" microcalorimeter (~10 eV FWHM for real-time analog processing over the energy range 0 keV to ~10 keV) allows straightforward separation of closely spaced x-ray peaks in important materials (such as TiN and WSi_2), which cannot be resolved by semiconductor EDS.

We have recently fabricated a microcalorimeter with an instrument-response energy resolution of 3.1 eV ± 0.1 eV FWHM (digital processing) and ~4 eV FWHM (analog processing) over the energy range 0 keV to ~2 keV.[2] In Fig. 1, we show a microcalorimeter EDS x-ray spectrum of a 0.3-μm-diameter Al_2O_3 particle on a Si substrate. In Fig. 2(a), we present x-ray spectra of Fe and FeO-OH, demonstrating the new capability of the microcalorimeter EDS for chemical shift measurements.[3] The observed chemical shifts in the Fe-L x-ray lines are identical to that obtained using a WDS on the same specimens, shown in Fig. 2(b).

In Fig. 3, we summarize the energy resolution as a function of energy for our 3 eV (circles) and 10 eV (squares) detectors, semiconductor EDS, and WDS. The resolution of microcalorimeter EDS is more than an order of magnitude higher than that of semiconductor EDS and surpasses even that of WDS at some energies. With an effective area within a factor of 2.5 and speed within a factor of 3 of high-resolution semiconductor EDS, we believe that microcalorimeter EDS will provide revolutionary new capabilities for x-ray microanalysis.

References

1. D. A. Wollman *et al.*, *J. Microscopy*, 188 (1997) 196.
2. K. D. Irwin *et al.*, in preparation.
3. D. A. Wollman *et al.*, *Proceeding of Microscopy and Microanalysis '98* Atlanta (1998).
4. Contribution of the U.S. Government; not subject to copyright. We acknowledge Alain Diebold and Benjamin Liu for providing the Al_2O_3 particle specimen.

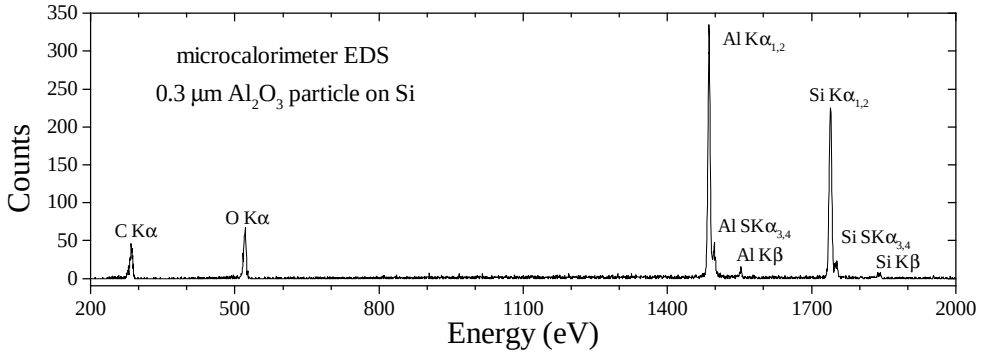

Fig. 1. Microcalorimeter EDS x-ray spectrum of a 0.3 μm Al_2O_3 particle on a Si substrate, acquired under the following conditions: 5 keV beam voltage, 40 pA beam current, spot mode, 200 s live time, 57 s^{-1} input count rate, 54 s^{-1} output count rate, 5% dead time, and an x-ray takeoff angle of 45°. The spectrum was corrected for energy nonlinearity, resulting in a nonuniform energy binwidth of ~0.65 eV over the energy range presented. A significant C contamination peak developed during the acquisition of the spectrum.

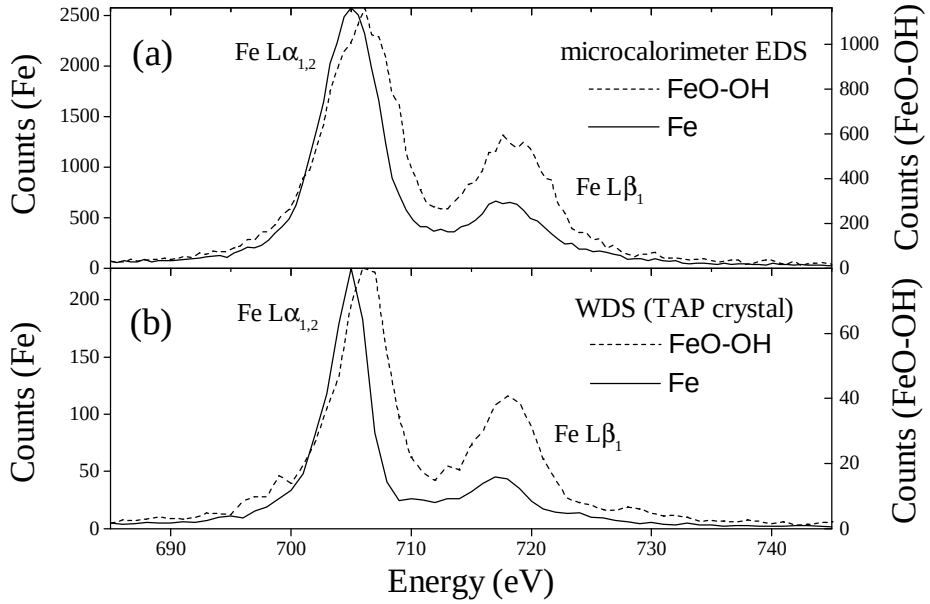

Fig. 2. (a) Microcalorimeter EDS spectra and (b) WDS spectra of Fe (solid line) and FeO-OH (dashed line), from Ref. 3. The observed changes in the Fe-L peak positions and intensities result from chemical bonding effects.

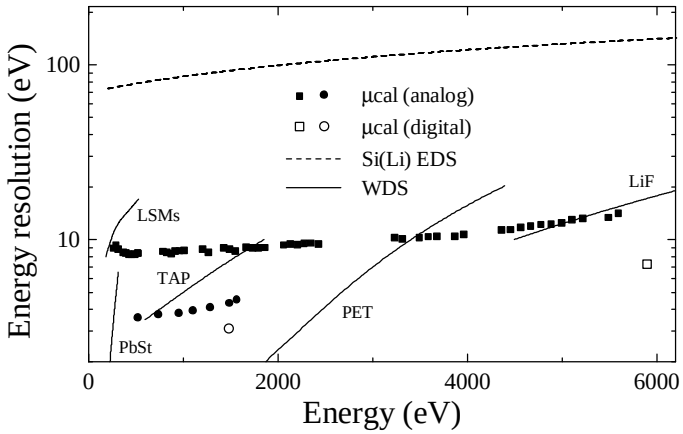

Fig. 3. Energy resolution vs. energy for microcalorimeter EDS (μcal EDS, analog and digital processing), semiconductor EDS, and WDS, after Ref. 1. The Si(Li) EDS energy resolution is 140 eV at 5.9 keV, and the WDS crystals are lithium fluoride (LiF), pentaerythritol (PET), thallium acid phthalate (TAP), lead stearate (PbSt), and layered synthetic materials (LSMs).